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SPECIFICATION FOR LIMESTONE FOR CHEMICAL INDUSTRY

(First Revision)

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Indian Standard
SPECIFICATION FOR LIMESTONE FOR CHEMICAL INDUSTRY
(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 4 August 1978, after the draft finalized by the Inorganic Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first issued in 1965; in this revision changes have been made in the methods of determination of manganese, sulphur and phosphorus in the light of experience gained during these years.

0.3 Limestone is an important basic raw material for the chemical industry. It is widely distributed throughout India. Almost every state possesses limestone deposits of various degrees of purity. The chemical industry uses relatively small quantities of limestone as compared to quicklime and hydrated lime, although many industries can use both these materials.

0.4 Physical requirements, such as specific gravity, porosity, compressive strength and size of stone are not included in this standard, as these requirements will depend on, amongst others, the types of kilns used for burning limestone. The Sectional Committee responsible for the preparation of standard, therefore, decided that these requirements shall be subject to agreement between the purchaser and the supplier.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for limestone for chemical industry. It also covers sea-shells and calcite, a crystalline form of naturally occurring calcium carbonate.

1.1.1 This standard does not cover limestone for building, agricultural, metallurgical, glass and ceramic industries.

2. GRADES

2.1 The limestone shall be of the following four grades depending upon their suitability for various uses on the basis of their chemical properties:

Grade 1 — suitable for the manufacture of bleaching powder, bleach liquor, textiles, varnishes, pulp and paper.

Grade 2 — suitable for the manufacture of soda ash by the Solvay process, caustic soda by the lime-soda process.

Grade 3 — suitable for the manufacture of calcium carbide.

Grade 4 — suitable for sugar manufacture.

3. REQUIREMENTS

3.1 Description — The material shall be in the form of lumps, free from dirt and added impurities.

3.2 The material shall comply with the requirements given in Table 1 when tested according to the methods prescribed in IS : 1760-1962* and in Appendix A of this standard. Reference to the relevant test methods is given in col 7 and 8 of the table.

4. PACKING AND MARKING

4.1 Packing — The material shall be supplied in open wagons, or as agreed to between the purchaser and the supplier.

4.2 Marking — A metallic or cardboard lebel of appropriate size bearing the following information with suitable paint or ink shall be conspicuously displayed on the carrier and also placed inside:

a) Name and grade of the material;
b) Name of the supplier and his recognized trade-mark, if any;
c) Gross and net mass;
d) Batch number; and
e) Date of supply.

---

*Methods of chemical analysis of limestone, dolomite and allied materials.

TABLE 1 REQUIREMENTS FOR LIMESTONE FOR CHEMICAL INDUSTRY

(*Clauses* 3.2 *and* 5.2.2)

SL No.	CHARACTERISTIC	REQUIREMENT FOR				METHOD OF TEST, REF TO CL NO. IN	
		Grade 1	Grade 2	Grade 3	Grade 4	Appendix A	IS : 1760-1962*
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	Loss on ignition, percent by mass, *Max*	46·0	46·0	46·0	44·0	—	5
ii)	Silica (as SiO_2), percent by mass, *Max*	0·75	—	1·0	2·0	—	6
iii)	Iron (as Fe_2O_3), percent by mass, *Max*	0·15	—	0·25	—	—	8
iv)	Calcium (as CaO), percent by mass, *Min*	54·0	53·0	54·0	50·0	—	9
v)	Magnesium (as MgO), percent by mass, *Max*	2·0	1·0	0·8	1·0	—	10
vi)	Manganese (as Mn_2O_3), percent by mass, *Max*	0·06	—	—	—	A-2	—
vii)	Carbon dioxide (as CO_2), percent by mass, *Min*	42·0	42·0	42·0	41·0	—	13
viii)	Sulphur (as S), percent by mass, *Max*	—	—	0·1	—	A-3	—
ix)	Phosphorus (as P), percent by mass, *Max*	—	—	0·01	—	A-4	—
x)	Alumina (as Al_2O_3) and ferric oxide as (Fe_2O_3) together, percent by mass, *Max*	—	—	0·5	1·5		7
xi)	Silica (as SiO_2), alumina (as Al_2O_3), and ferric oxide (as Fe_2O_3) together, percent by mass, *Max*	—	3·0	—	—	—	6 and 7

*Methods of chemical analysis of limestone, dolomite and allied materials.

4.3 BIS Certification Marking

The product may also be marked with Standard Mark.

4.3.1 The use of the Standard Mark is governed by the provisions of th Bureau of Indian Standards Act, 1986 and the Rules and Regulations mad thereunder. The details of conditions under which the licence for the use o Standard Mark may be granted to manufacturers or producers may be obtaine from the Bureau of Indian Standards.

5. SAMPLING

5.1 Preparation of Test Samples — The method for drawing representative samples of the material shall be as prescribed in IS : 2109-1962*.

5.2 Number of Tests

5.2.1 Tests for determination of loss on ignition, silica and calcium shall be carried out on each of the individual samples.

5.2.2 Tests for the determination of the remaining characteristics given in Table 1 shall be performed on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples* — For those characteristics which are tested on individual samples, the mean and the range of test results shall be computed as follows:

$$\text{Mean } (\bar{X}) = \frac{\text{Sum of individual test results}}{\text{Number of test results}}$$

$$\text{Range } (R) = \text{Difference between the maximum and the minimum values of test results}$$

For declaring the conformity of the lot:

$\bar{X} + 0{\cdot}6\ R$ shall be less than or equal to the maximum specified requirement, or

$\bar{X} - 0{\cdot}6\ R$ shall be greater than or equal to the minimum specified requirement.

5.3.2 *For Composite Sample* — For declaring the conformity of the lot to the requirements of all characteristics tested on the composite sample, the test results shall satisfy the corresponding specified requirements.

APPENDIX A

(*Clause* 3.2 and *Table* 1)

METHODS OF TEST FOR LIMESTONE FOR CHEMICAL INDUSTRY

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

---

*Methods of sampling dolomite, limestone and other allied materials.

†Specification for water for general laboratory use (*second revision*).

A-2. MANGANESE

A-2.0 Outline of the Method — Manganese is determined colorimetrically by visual comparison.

A-2.1 Apparatus

A-2.1.1 *Platinum Crucible*

A-2.1.2 *Nessler Cylinders* — 100 ml capacity (*see* IS : 4161-1967*).

A-2.2 Reagents

A-2.2.1 *Dilute Nitric Acid* — 1 : 1 (*v/v*).

A-2.2.2 *Sodium Carbonate* — *See* IS : 296-1974†.

A-2.2.3 *Dilute Phosphoric Acid* — 1 : 1 (*v/v*).

A-2.2.4 *Potassium Periodate* — solid.

A-2.2.5 *Standard Manganese Solution* — Dissolve 0·1 g of pure electrolytic grade of manganese in dilute nitric acid, boil off nitrous fumes and make up the volume to 1 000 ml mark in a volumetric flask. One millilitre of this solution is equivalent to 0·1 mg of manganese (as Mn).

A-2.3 Procedure — Weigh accurately about 1 g of the material and dissolve in about 25 ml of dilute nitric acid. Filter and wash the residue with hot water. Ignite the residue in a platinum crucible, fuse with a little sodium carbonate, dissolve in dilute nitric acid and add to the main filtrate. To the solution (if necessary, filtered) add 20 ml of dilute phosphoric acid and 0·6 to 0·8 g of potassium periodate. Heat the solution to boiling and keep at boiling point for about 10 minutes (one hour for very small amounts). Cool the solution to room temperature. Transfer the solution completely to a Nessler cylinder and make up the volume to 100 ml mark. Simultaneously take 20 ml of dilute phosphoric acid and 0·6 to 0·8 g of potassium periodate in another beaker and add 4·2 ml of standard manganese solution. Heat the solution and boil as above. Cool and transfer to another Nessler cylinder and make up the volume to 100 ml.

A-2.4 The material shall be deemed to have passed the requirement of the test if the colour produced in the test with the material is equal to or less than that produced in the control test.

---

*Specification for Nessler cylinders.

†Specification for sodium carbonate, anhydrous (*second revision*).

A-3. SULPHUR

A-3.0 Outline of the Method — Sulphur is oxidized with bromine water and then precipitated as barium sulphate by addition of barium chloride solution.

A-3.1 Apparatus

A-3.1.1 *Platinum Crucible*

A-3.1.2 *Muffle Furnace*

A-3.2 Reagents

A-3.2.1 *Sodium Carbonate* — *See* IS : 296-1974*.

A-3.2.2 *Bromine Water* — saturated solution.

A-3.2.3 *Dilute Hydrochloric Acid* — 1 : 1 (*v/v*).

A-3.2.4 *Methyl Red Indicator Solution* — Dissolve 0·15 g of methyl red in 500 ml of water.

A-3.2.5 *Dilute Ammonium Hydroxide* — 1 : 1 (*v/v*).

A-3.2.6 *Barium Chloride Solution* — 10 percent (*m/v*).

A-3.3 Procedure — Weigh accurately about 2·5 g of the finely powdered material in a platinum crucible and mix it with 10 g of sodium carbonate. Place the crucible in a furnace and gradually raise the temperature of the furnace to 800°C. Keep the crucible for half an hour at this temperature and finally raise to 1 000°C for fusion. Cool and place the crucible in a 250-ml beaker and cover with hot water. Boil till the melt breaks down completely to powder. If necessary, hasten the process by crushing any lumps with a glass rod. When no lumps are left, filter through a fast filter paper and wash well with hot water containing about 1 percent sodium carbonate. Reject the residue and evaporate the total filtrate to about 150 ml, after adding 10 ml of bromine water and 30 ml of dilute hydrochloric acid. During this all excess bromine will be expelled. Add a few drops of methyl red indicator and make it alkaline with dilute ammonium hydroxide. Boil and filter, washing with hot water. To the filtrate add 5 ml of dilute hydrochloric acid, dilute to about 200 ml and boil. To the boiling solution, add slowly, with constant stirring, 10 ml of hot barium chloride solution. Allow the precipitate to stand overnight. Filter through a filter paper or Gooch crucible and ignite at 800°C to constant mass.

A-3.3.1 Carry out a blank determination simultaneously using the same quantities of all the reagents.

---

*Specification for sodium carbonate, anhydrous (*second revision*).

A-3.4 Calculation

$$\text{Sulphur, percent by mass} = \frac{13\cdot74 \times (M_1 - M_2)}{M}$$

where

M_1 = mass in g of the barium sulphate precipitate obtained from the material,

M_2 = mass in g of the barium sulphate precipitate obtained from the blank, and

M = mass in g of the material taken for the test.

A-4. PHOSPHORUS

A-4.1 Dissolve 2 g of the material in 20 ml of dilute hydrochloric acid (1 : 1, *v/v*) with a few drops of concentrated nitric acid. Filter and proceed with the filtrate according to method prescribed in **A-4** of IS : 574-1970*.

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*Specification for trisodium phosphate (*first revision*).

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Headquarters:

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